WISCONSIN DEPT. OF NATURAL RESOURCES

LABNOTES
Spring 1998

The newsletter of the Wisconsin Laboratory Certification and Registration Program
Program Information: (608) 267-7633 Telefax: (608) 266-5226

NR Board approves rule change, fees for FY 1999

Jeffrey Ripp, Lab Certification Program

Changes in state rules governing lab certification and registration will take effect on June 1, barring unanticipated legislative action. The changes to NR 149, Wis. Adm. Code, were approved by the Natural Resources Board in February. The changes will clarify the chapter, add new definitions and expand the list of analytical method sources. The NR Board also approved amendments that will adjust the fees for certified labs, move several tests to new categories and create a new test category for immunoassays. Currently, the rule changes are being reviewed by the legislature. If no additional hearings are held, the rules will take effect as proposed and Wisconsin certified and registered laboratories need to begin making the necessary adjustments for compliance.

Fees. The new rule adjusts the relative value of several fee items. The amendments will increase the base fee for certified laboratories from 10 relative value units (RVU) to 15 relative value units. The base fee for registered laboratories remains the same as before. The fees for laboratories accepted under reciprocity agreements will increase from 24 to 30 RVU. Also, the fees for laboratories certified only for nitrate and fluoride in category 18 (safe drinking water) are reduced from 20 to 4 RVU. Laboratories will notice this change in their renewal bills which will be mailed in late May of this year.

Tests. The new rule makes several changes in tests and test categories. Sulfide will be moved from category 5 to category 6. Glycols will be added as a test in category 10. Explosive residues, n-methyl carbamate pesticides and substituted urea pesticides will be created as tests in category 13. The rule also creates a new test category for immunoassay tests. This category has a delayed implementation date and will not be offered until September 1999. See page 3 for more information about applying for the new tests.
(Please see NR Board on page 2)

IN THIS ISSUE....

LabNotes - Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

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NR Board, from page 1

BOD. The new rule requires laboratories to analyze a known standard for biochemical oxygen demand (BOD) after every 20 samples, or at a minimum of once per week if fewer than 20 samples are analyzed. This change will affect all laboratories, including small wastewater treatment plants.

Copies of the rule are available by contacting the Laboratory Certification and Registration Program at (608) 267-7633 or for free on the following web site:

http://www.legis.state.wi.us/rsb/code/

FEES FOR FY 1999

In a related action, the NR Board approved the fees for the Laboratory Certification and Registration Program at the March 1998 meeting. The Board approved keeping the fees at the same level as last year, with the cost-per-relative value unit (RVU) at $37.50. Registered laboratories will not see any change in their bill for FY 1999 but certified and reciprocity laboratories will see an increase in their annual fee as a result of the changes to chapter NR 149. The base fee for certified laboratories will increase from 10 to 15 relative value units. All certified laboratories should expect a $187.50 increase over last year's fees. The DNR is planning to mail out the bills in mid-May. For more information about the laboratory certification and registration fees, please contact Jeff Ripp at (608) 267-0579 or by e-mail at *rippj@dnr.state.wi.us.*

Laboratory Fees for FY 1999 (Sept. 1, 1998 - Aug. 30, 1999)			
Fee Item	**Unit Price FY 1999**	**Fee Item**	**Unit Price FY 1999**
Registered Base Fee	$375.00	Category 11	$150.00
Certified Base Fee	$562.50	Category 12	$150.00
Initial Application Fee	$225.00	Category 13	$150.00
Revised Application	$112.50	Category 14	$150.00
Category 1	$37.50	Category 15	$450.00
Category 2	$37.50	Category 16	$150.00
Category 3	$37.50	Category 17	$150.00
Category 4	$37.50	Category 18	$750.00
Category 5	$75.00	Cat. 18 (Nitrate Only)	$75.00
Category 6	$75.00	Cat. 18 (Nitrate + Fl. Only)	$150.00
Category 7	$150.00	Category 19	$150.00
Category 8	$150.00	Category 20	$975.00
Category 9	$150.00	Category 21	$150.00
Category 10	$150.00	Reciprocity	$1125.00

New tests, test categories to take effect July 1, 1998

Jeffrey Ripp, Lab Certification Program

The amendments to chapter NR 149 will move tests, create new categories and add new tests to old categories. Questions about these changes should be addressed to Jeff Ripp at (608) 267-0579 or by e-mail at *rippj@dnr.state.wi.us.*

Category 6 - Sulfide: Sulfide was moved from category 5 to category 6 to reside with its hazardous waste testing cousin, cyanide. The Department will make this change for laboratories that are currently certified or registered for sulfide. If your lab is currently certified or registered for sulfide, your 1999 certificate will reflect the change. Laboratories that wish to become certified or registered for this test must submit a revised application. Reference samples are not required for sulfide.

Category 10 - Glycols: A new test was created in category 10 for glycols and other nonhalogenated volatiles using SW-846 method 8015. Laboratories interested in performing this test will be required to submit an application for certification or registration. The Department will begin accepting applications for this test after July 1, 1998. Reference samples are not required for glycols.

Category 13 - n-Methyl Carbamates and Substituted Urea pesticides: These tests were created to recognize the difference between SW-846 methods 8318 (n-methyl carbamates) and 8321 (substituted urea pesticides). Laboratories that are currently certified or registered for carbamate pesticides in category 13 will have these tests automatically added to their certificates. Laboratories that wish to obtain certification or registration for these tests must submit a revised application form. Reference samples are required for these tests, however one sample is sufficient for both tests.

(Please see new tests on page 4)

New Tests, from page 3

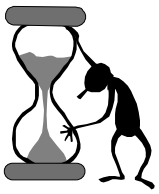
Category 13 - Explosive Residue by LC: Several laboratories have requested certification for explosive residues by liquid chromatography techniques. Most labs are using SW-846 method 8330. Laboratories that are currently certified for this test under category 19 will have this test moved to category 13. Other laboratories that wish to obtain certification for this test will need to submit a revised application. Reference samples are required for this test. The Department currently accepts explosive residue samples from several providers.

Category 21 - Immunoassay Testing: The Department created a new test category for laboratories performing immunoassay work for a covered program. This category will not be effective until September 1, 1999. Between now and December 1998, the Department will develop an implementation plan for this category. The Department expects to begin accepting applications in 1999. More information about this test category will be included in the Fall 1998 issue of *LabNotes.* If you think that your lab would be interested in applying for this category, we would be interested in hearing your comments and suggestions. Please contact Jeff Ripp at (608) 267-0579 or by e-mail at *rippj@dnr.state.wi.us.*

Spring means renewal time in Wisconsin

January	• DNR begins accepting reference sample results.
May	• DNR sends a letter requesting reference sample results. • DNR mails renewal bills to labs.
June	• DNR begins mailing renewal certificates to labs that have paid their renewal fees and submitted acceptable reference sample results.
August	• DNR assesses a late fee to labs which have not paid their renewal bill by August 1.
September	• Renewal period ends September 1. All fees and references samples are due. Labs that have not submitted fees and reference samples will not be renewed after Sept. 1.

Renewal time is here again for the Wisconsin Laboratory Certification and Registration Program. This year is particularly special: For the first time in five years, all of our certified and registered labs will be on the same renewal cycle. Your laboratory's certificate will be sent out between June and August and will expire on August 31, 1999. This means that all laboratories should receive a new certificate before their current one expires. In past years, there often was a lag time from when your certificate expired and when you received your new one. We are very excited to provide this service and anticipate a seamless transition period. To make sure that renewal goes smoothly for your laboratory, the above table contains deadlines that you should keep in mind. If you have any questions about renewal, please contact Mike Kvitrud at (608) 261-8459 or by e-mail at *kvitrm@dnr.state.wi.us.*



SW-846 method 5035 will not be used in Wisconsin

Greg Pils, Lab Certification Program

With the release of SW-846 Update III late last year, our office has received many calls from laboratories asking if Wisconsin now requires the use of method 5035 for the analysis of volatile organic compounds (VOCs) in soil and waste samples. The answer is a definite "No." All labs performing this type of testing must adhere to the requirements set by section 700.13 - sample preservation and analysis, in chapter NR 700, Wisconsin Administrative Code. Required sampling and analysis procedures for VOCs are addressed in subsection (2), which states:

(2) VOLATILE ORGANIC COMPOUNDS. (a) Soil samples collected for analysis of volatile organic compounds for compliance with chs. NR 700 to 736 shall be preserved in methanol immediately after collection unless the samples are stored in a device which insures sample integrity. Samples stored in a suitable device, including brass tubes and En Core™ samplers, shall be preserved in methanol according to sub. (3). The Department may approve alternate storage devices on a case-by-case basis prior to use in the field. Samples shall be preserved and handled as specified in section 8 and Table 1 of the "Modified GRO, Method for Determining Gasoline Range Organics [September 1995 revision]". (b)

Methanol-preserved soil samples shall be extracted in the vial by shaking for 2 minutes and sonicating in an ultrasonic water bath for 20 minutes. After sonication, an aliquot not larger than 100 microliters shall be removed and introduced into a purge and trap system as in par. 7.3.3.2.4 of method 5030A (note: now 5030B) in "Test Methods for Evaluating Solid Waste (SW-846)." (c)

Methanol-preserved soil samples shall be analyzed by gas chromatography or gas chromatography/mass spectrometry using capillary columns. Suitable analytical methods are found in SW-846.

In other words, nothing has changed. Those concerned about future requirements can rest easy -- there are no immediate plans for incorporation of method 5035 into the Wisconsin certification and registration program. For more information, contact Greg Pils by phone at (608) 267-9564 or by e-mail at *pilsg@dnr.state.wi.us.*

DNR Environmental Achievement Awards

Has your company recently implemented one or more of these strategies to reduce pollution?

- improved operating practices
- material substitutions
- product changes
- technology changes
- closed loop recycling

If so, the DNR would like to celebrate your success! Your recent accomplishments could be the basis for the DNR's **Prevention-Environment-Prosperity (P/E/P) Award**. Contact Leah Nell Adams at (608)264-8951 or by e-mail at *adamsl@dnr.state.wi.us* for a nomination package.

NR Board approves new health-based groundwater standards

Steve Karklins, Bureau of Drinking Water and Groundwater

On March 25, 1998, the Natural Resources Board approved new health-based groundwater quality standards for 19 substances. The Board also approved revising the existing health based standard for cyanazine. After further consideration, the Board approved health based standards for both ammonia and boron at the April 29, 1998 meeting. The new standards added to NR 140 include: ammonia; anthracene; bentazon; benzo(b)fluoranthene; boron; carbon disulfide; chrysene; cobalt; dibutyl phthalate; fluoranthene; n-hexane; hydrogen sulfide; methanol; n-nitrosodiphenylamine; prometon; pyrene; pyridine; 1,1,1,2-tetrachloroethane; 1,2,3-trichloropropane; trimethylbenzenes (1,2,4 - and 1,3,5- combined); and vanadium.

The NR 140 rule package has been forwarded to the legislature for consideration. After approval the rule will move on to the Revisor of Statutes for promulgation. The Department anticipates promulgation of the standards sometime this fall; barring legislative hearings.

Contact Steve Karklins in the Department's Bureau of Drinking Water and Groundwater at (608)266-5240 or by e-mail at *karkls@dnr.state.wi.us* for additional information.

REMINDER: The deadline for Wisconsin certification/registration renewal is September 1, 1998. Make sure you have paid the fees and submitted reference samples!!

DNR introduces new wastewater permit application process

Tom Mugan, Bureau of Watershed Management

Starting in the latter half of 1998, look for changes in the look and feel of permit applications and required testing for WPDES facilities. DNR is completing work on a whole new application which will eventually allow electronic submissions but in the short term is intended to do a better job of collecting the right analytical information.

"We are trying to provide an early warning system to determine when we need to verify detects of toxic chemicals very near the detection limit or when multiple sample results are needed for statistics-based decisions," says Tom Mugan of the Department's Permit Coordination Section. In the past, the Department placed regulatory requirements in permits based on as little as a single detect for a toxic substance.

The new application was the topic of a DNR workshop on March 19 attended by laboratory representatives and engineering consultants. "The Department believes getting lab personnel more directly involved in the information loop will improve our ability to make good science-based decisions," Mugan said. The Department hopes to hold similar information sharing sessions in the future to find out how well the new application is working.

One part of the new application format is the section on preliminary limits. Information provided by preliminary limits will help permittees (and their labs and consultants) know if they used a method sensitive enough for a given pollutant measurement,

(Please see Permit Application on page 7)

Permit Application, from page 6

or if based on initial results, they should collect additional samples for testing.

Certain substances which are toxic at low doses, such as pesticides, may require special care in evaluating and reporting results. Application instructions state that all detects for organics must be confirmed. Results from double column analysis are not always definitive. In some past situations, numerical results for substances initially thought to be present were later found to fall beneath a matrix-specific method detection limit (MDL).

The Department is emphasizing that permittees should share certain parts of the permit application packet with their contractor labs. This will help the labs do their work more effectively and make the process go more smoothly for everyone. Please let us know how well you think it's working. Contact Tom Mugan with your comments, suggestions and, yes, gripes. Tom's phone number is (608) 266-7420 and e-mail is *mugant@dnr.state.wi.us.*

1998 Lab of the Year Award Winners. From left: Al Kardoske, De Pere WWTP, Jack Sullivan, WDNR and Tad Schwartzhoff, Dairyland Power.

Dairyland Power Cooperative, De Pere Wastewater Treatment Plant honored by NR Board

Jeffrey Ripp, Lab Certification Program

The Dairyland Power Cooperative Environmental Laboratory and the De Pere Wastewater Treatment Facility received the 1998 Registered Laboratory of the Year awards at the March 1998 Natural Resources Board meeting. The awards program began in 1996 and is intended to recognize those laboratories that have developed exceptional systems for producing high quality data.

Dairyland Power Cooperative's Environmental Laboratory in La Crosse was chosen to receive the award for the large registered facility. This facility provides analytical service to the Cooperative's generating facilities and performs a wide range of chemical tests in air, wastewater, groundwater, and hazardous waste samples. The laboratory passed its most recent on-site evaluation in September 1997 without any major deficiencies.

Rick Mealy, lab auditor for the DNR, commended the facility for its dedication and cooperation during the audit. "This was the very first instance in which I granted registration before issuing the audit report because I was so impressed by the lab's commitment to producing quality results," said Mealy.

The laboratory performs many complex test procedures extremely well, Mealy said. For example, he found no major deficiencies with the toxicity characteristic leachate procedure, a complicated hazardous waste test that many laboratories have difficulty performing correctly.

(Please see Lab of the Year on page 8)

Lab of the Year, from page 7

Dairyland has developed a top-notch system for tracking analytical results and monitoring performance. The laboratory has created individual binders for each analytical test. These binders contain the lab's standard operating procedures, method references, information about standards and reagents, blank bench sheets, completed bench sheets, quality control charts and calibration information. All of the pertinent details of a test are included in these binders, making verification of analytical results easy.

The **City of De Pere Wastewater Treatment Plant Laboratory** was chosen to receive the award for the small registered facility. This lab provides analytical support for the wastewater treatment plant and analyzes wastewater samples for biochemical oxygen demand, ammonia, total suspended solids and phosphorus. The facility has a consistent record of compliance with strict permit limits. This can be partially attributed to the excellent work performed in the plant's laboratory. The laboratory continues to receive high marks from its regional auditor and area engineer. The facility was last evaluated in September 1996.

According to Linda Vogen, the lab's auditor, "The wastewater laboratory at De Pere exemplifies what was intended when the laboratory certification program was developed. The staff of the lab not only carry out the requirements of NR 149 but make maximum use of the information generated."

De Pere uses a computerized system to calculate control limits, track quality control compliance and perform linear regression analyses for calibrations. Based on this information, the analysts have the freedom to make the necessary adjustments in sample sizes, re-analyze out of control samples, and interpret results. The laboratory is well organized and diligently documents the necessary information for verifying analytical results, including equipment maintenance records, calibration information, standard operating procedures and results of quality control samples.

These laboratories were chosen from a strong pool of nominees, making selection of the recipients difficult. Other laboratories nominated for these awards were the Fontana-Walworth Water Pollution Control Facility, the City of Stanley Wastewater Treatment Facility, and the City of Two Rivers Wastewater Treatment Facility. Congratulations to all of the nominees, and especially to the Dairyland Power Cooperative and the De Pere Wastewater Treatment Facility.

Nominations for the 1999 Registered Laboratory of the Year awards are open to anyone, including DNR staff. Nominations are due by December 31, 1998. Contact Jeff Ripp at (608) 267-0579 or by e-mail at *rippj@dnr.state.wi.us* for more information or to obtain a nomination form.

Advisory group welcomes new member

Agricultural representative William Bruins is the newest member of the Certification Standards Review Council. Mr. Bruins operates a dairy farm near Waupun. The council is a nine member advisory body to the Wisconsin DNR. Members represent a diverse group of environmental interests throughout the state including municipal, industrial and commercial laboratories. The council makes recommendations concerning the certification program. For more information on the council, its membership or its meetings, please visit the DNR's web site at:

http://www.dnr.state.wi.us/org/es/science/lc

Reference sample news

Mike Kvitrud, Lab Certification Program

EPA reference sample (PE) study update

We have heard for quite some time that EPA will no longer be providing the Water Supply (WS), Water Pollution (WP) or the Discharge Monitoring Quality Assurance (DMR-QA) PE Studies after 1998.

So what will labs need to do now?

EPA intends to approve private companies and local agencies to supply the samples at a cost to labs. These private companies and local agencies will, in turn, forward the results to EPA. Under this plan, your lab could use any EPA-approved PE provider for compliance. The results will be forwarded to the EPA and other accrediting agencies, including Wisconsin, for renewal.

How will EPA accomplish this?

EPA is still working out the details of the implementation plan. Watch for more information in the Fall 1998 *LabNotes.*

How will this affect your lab?

Nothing will change for renewing your Wisconsin non-drinking water (SDWA) certification or registration. Wisconsin already accepts PE results from many different providers including Analytical Products Group (APG), Analytical Standards Inc. (ASI), Environmental Resource Associates (ERA), the Wisconsin State Laboratory of Hygiene (SLH) and the New York Department of Health (NYDOH).

What about drinking water?

As for your Wisconsin drinking water (SDWA) certification, there will be more options available to you. In the past, only EPA's WS study was acceptable for SDWA certifications. Starting next year, Wisconsin will accept other water supply studies as long as the provider uses the same grading criteria (fixed limits) as the EPA WS study. These limits can be found at 40 CFR Part 141.23 and 141.24.

To wait or not to wait, that is the reference sample question

We have been noticing that many labs are relying on fixed limit reference (PE) samples for obtaining or renewing their certifications or registrations. From our perspective, these samples may disadvantage your lab and should only be used when your lab needs to quickly show that it has fixed a problem and come back into compliance. What are fixed limit PE samples and why do we discourage their use? Fixed limit studies are PE studies in which only a small number of labs participate. These are sometimes called quick response or quick turn around samples and have fixed acceptance limits which are usually narrower than the acceptance limits of a regular PE study. This can be a disadvantage if there is a problem with the sample or if the sample has a high or low recovery bias.

The alternative to fixed limit reference samples is to participate in peer-graded reference sample (PE) studies. By participating in these studies, a lab is being compared with other laboratories. One advantage

(Please see Reference Samples on page 10)

Reference Samples from page 10

is that if there is a problem with the sample or if there is a high or low recovery bias, all laboratories will have the same problem. The acceptance limits will be adjusted to reflect the sample's difficulty.

Peer-graded studies are offered surprisingly often, and most labs should have no problem finding a schedule that works for them.

It is better to have participated and failed then to have never participated at all

We have been receiving a lot of questions lately about when labs "have to" participate in the next reference sample (PE) study in order to renew their Wisconsin certification or registration. The easy answer to this question is that a lab only needs to pass one PE sample for each of its certified or registered tests and test categories between January 1 and August 31 of each year. But our question in return is "Why wouldn't you want to participate in more?"

Participating in several PE studies will not jeopardize your Wisconsin certification or registration unless your lab is consistently reporting unacceptable results. Please do not refrain from participating in more than one PE study simply because you may "fail". Having one unacceptable result when you have acceptable results in other studies does not put a blemish on your certification or registration status.

One, two, three strikes, you're out!

Now that we have just encouraged you to participate in more than one reference sample (PE) study, we need to inform you of the consequences of consistent reference sample (PE) failure. As stated in the previous section, one unacceptable PE result among other acceptable ones is not a reason for concern. On the other hand, a lab that consistently has unacceptable results for a specific parameter probably has a systematic problem affecting data quality. When this happens, the DNR will request two things from the lab: 1) notification that the lab is looking into and correcting the problem and 2) proof that the lab has corrected the problem in the form of acceptable PE results. When these two items are not supplied in a timely fashion, the Department may initiate enforcement actions against the lab. The Department may suspend a laboratory's certification for reporting three consecutive unacceptable results for a specific parameter. In drinking water (SDWA), the Department may revoke a lab's certification for two consecutive unacceptable results. If you would like more information about the DNR's policy concerning consecutive reference sample failure, please contact Jeff Ripp at (608) 267-0579 or by e-mail at *rippj@dnr.state.wi.us.*

Reminder: turn in those surveys!

We have not received everyone's response to the LOD information request survey that was sent out this past January. If your lab has not returned the diskette yet, please do so immediately. If you are unable to submit the requested information on the diskette that was enclosed, please send a paper copy. The information will be compiled and used to estimate the range and variability of the LODs and LOQs used by labs in the Wisconsin program. This information will also be used to determine compliance with the low-level data reporting requirement. Contact Greg Pils at (608) 267-9564 for more information.

The Auditor's Corner

Alfredo Sotomayor, Senior Audit Chemist

SALMAGUNDI

This year the Laboratory Certification and Registration Program will be twelve years old. That is about four generations, in laboratory years. I had reason to ponder this recently when I audited a laboratory that I had first visited during my initial rounds in 1987. Shifts in the regulatory climate and the economy have not been kind lately to the laboratory community and particularly, to commercial laboratories. So many changes...and so many more are predicted. Few of the laboratory directors, quality assurance officers, and analytical staff that I came to know and deal with at the inception of the program are still in the same laboratories where I first encountered them. Many have switched careers. "Nostalgia Isn't What It Used to Be Anymore," the title of Simone Signoret's biography, defines my mental state as I randomly look back in wonder.

"A national accreditation program will not be a reality until the year 2000". That seemed so far away in 1987, when I first made the comment. Well, the millennium is approaching, NELAC standards have been approved, and maybe by 1999 there will be laboratories bearing the NELAP insignia. Not too bad, I say, for an off-the-cuff pronouncement. Some are predicting that many years from now all data archeologically retrieved will be identified by two flags, BN and AN, for before and after NELAC; but some people do get carried away with predictions.

Although fashion cyclically casts retrospective glances, few penetrate the collective wardrobe of society let alone the closets of chemists. Technology is less prone to romanticize the past. In environmental analysis the allure of the new is very powerful. Instruments promising more convenience, more accurate results, and improved sensitivity tend to irreversibly displace the old and cumbersome. Here is a partial and personal list of items and practices that have suffered or are destined to suffer the same fate as leisure suits, retro-trendiness notwithstanding: Packed columns; hand-drawn calibration curves; analog analytical balances; gaseous hydride analysis; plotting control charts by hand; strip-chart recorders; chelation extraction analysis of metals; direct current plasma spectrophotometers; freon extractions for oil and grease; needle-scale spectrophotometers; preparing stock metal solutions from salts; EP Toxicity tests; cutting and weighing chromatograms for quantitative analysis.

Did you know or do you remember that there was an approved procedure for analyzing trihalomethanes in drinking water, Method 501.3, by selective ion monitoring (SIM) mass spectrometry? Curiously, years after this method was retired, there is now an allowance for using SIM in most SW-846 GC/MS methods.

A decade ago mass spectrometry was still the province of academic or research institutions and laboratories with big budgets or within the CLP. In those days, GC/MS operators were very much in demand and commanded what seemed then impressive salaries. Today, GC/MS instruments are ubiquitous, affordable, and much more sensitive. As for the demand for GC/MS operators and their salaries, let us just say that the market has once again mercilessly adjusted itself.

(Please see Auditor's Corner on page 12)

Auditor's Corner, from page 11

It is sad to witness the erosion of fundamental skills that used to be universal in chemistry laboratories: Transferring or delivering liquids using pipets; reading the volume of a serological pipet; decanting techniques; matching spectrophotometric cuvettes; standardizing with primary standards; performing error propagation analysis. I believe this is the result, in part, of increasing reliance on instrumental analysis, but I also suspect that analysts are not being trained in school or at work in these so called "old-fashioned" techniques. Many see wet chemistry as utterly unglamorous. In deemphasizing it and relegating it to the less competent, we neglect to consider how this is the traditional entry-point discipline for many analysts. Bad habits learned here will not only remain with an analyst for a long time, but will also make inroads into other laboratory operations.

Oh, how we hate BODs! This staple analysis, contrary to expectations, is one of the most difficult tests to master, yet we expect **everyone** to master it. Years ago I heard rumors that BOD would be replaced by other indicators of demand, that improved and direct assays would make it obsolete, or that automated determinations would make the test a lot easier. Alas for analysts and auditors, the BOD test is very much alive and kicking, and it has not gotten any easier. I do not think the millennium is going to eradicate the curse of BODs.

Computers have made data storage and manipulation a lot easier. Since this certification program started, the availability of computers and the proliferation of software specifically designed for analytical laboratories has been just short of miraculous. Some curious side-effects of this wonder treatment are notable. We find that some laboratories have data stored electronically but are not always able to retrieve it because the current software is incompatible with older versions. Those that attempt to reconstruct data from electronic archives end up at times with a data soup quite unlike the gourmet dish their palates remember. Until the millennium brings improved universal encoders and decoders, hard copy will likely remain essential, paradoxically owing its continued existence to the very agent that was to render it obsolete.

And to conclude this salad, I pose a question I've had for many years: Why does *Standard Methods* specify the concentration of solutions in Normality, but rarely gives the equations that govern analysis reactions? If you know, or are prone to speculate, would you let me know before the year 2000?

CORRECTION: Alfredo Sotomayor's phone number was incorrectly printed in the Fall 1997 issue of LabNotes. The correct number should be: (608) 266-9257. We regret the error.

Alternate Test Procedures: Laboratories interested in pursuing alternate test procedures (ATPs) with the United States Environmental Protection Agency should contact Ken Gunter at Region V, Chicago at (312) 353-9076.

PECFA Contact: Questions about PECFA *Update #13*, the request for the annual laboratory bids, should be addressed to Sheldon Schall, Bureau Chief for Storage Tank Regulation, Department of Commerce at (608) 266-0956.

Easy steps for obtaining registration for phosphorus

Susan Scobell Watson, Lab Certification Program
(reprinted from NW Ripplings, March 1998)

In light of changing permit requirements, quite a few labs are looking into doing their own phosphorus testing. Following the step-by-step procedures below will help those labs interested in applying for phosphorus certification or registration.

1. Select a DNR approved testing procedure by consulting chapter NR 219, Wis. Adm. Code. For example, *Standard Methods*, 18th Ed. 4500-P B & E or EPA "Methods for Chemical Analysis of Water and Wastes", 365.2.

2. Familiarize yourself with the requirements of the testing procedure. Look for techniques, principles, and interferences which are unique to this procedure.

3. Order the necessary equipment, glassware and chemicals including a phosphorus reference standard.

4. Create a data sheet to record sample volumes digested, standard curve data, absorbances for samples and standards, dilutions, calculations, etc. Refer to the DNR's "*Quality Assurance Document for a Small Wastewater Lab*" for benchsheets, spike analysis, and control limit calculations. [**ed. note:** The DNR is revising the QA manual for a WWTP. The new document should be available this summer.]

5. Practice, practice, practice the procedure until you are confident in your lab's ability to generate reliable data.

6. Perform a MDL (method detection limit) determination. Refer to the "*Wisconsin Laboratory Certification and Registration Program Information and Requirements"* booklet for more information on calculating MDLS. You can anticipate MDLs between 0.005 to 0.010 mg/L. Thus, the 7 replicates should be prepared at about 0.025 to 0.050 mg/L.

7. Set up quality control measures for phosphorus including: standard curve, digested blank, known standard, duplicates, spikes, interim QC acceptance limits, method references, etc.

8. Include the procedure with method references and quality control specifics in your laboratory's Quality Assurance Manual.

9. Analyze a reference sample successfully. Every year your lab will be required to submit reference sample results. Remember to order blind standards for phosphorus each year.

10. Submit a completed revised application form. To request an application form contact John Condron at (608) 267-2300.

The application will be processed and the laboratory will be audited within approximately 90 days. The audit may be waived at the discretion of the auditor. Upon successful completion of an audit or if the audit has been waived, the facility will be granted registration (or certification). As you can see, the whole process can take 4-6 months, so be sure to give yourself enough time to complete all the steps before you begin running your own samples to report on your DMR. For more information, contact John Condron at (608) 267-2300.

An overlooked requirement: thermometer calibrations

Susan Scobell Watson, Lab Certification Program (reprinted from NW Ripplings, March 1998)

The requirements for calibrating laboratory thermometers need clarification. *Standard Methods* allows temperature measurements with any good quality mercury-filled Celsius thermometer. At a minimum, the thermometer should have a scale marked for every 0.1° C. The markings must be etched on the capillary glass. The thermometer should be calibrated periodically against a precision thermometer certified by the National Institute of Standards and Technology. The thermometer must be used with its certificate and correction chart.

Wisconsin requires that thermometers be calibrated at least annually and tagged with any correction factor. If a lab has a thermometer that is certified by NIST, annual calibration is not necessary as long as the thermometer has not been damaged. The thermometer must be re-certified by NIST before the expiration date on the accompanying certificate. Similarly, annual calibration is not required if a lab has a thermometer that is factory certified by the manufacturer against an NIST certified thermometer. These thermometers also come with a certificate giving correction factors at specific temperatures. These thermometers do not need to be re-calibrated before the certificate expiration date unless there is some reason to suspect that they are not recording properly.

Of course, it is important to select thermometers that are certified at the temperatures that you intend to use them: 20° C for BOD incubators, 103° C for suspended solids ovens, and 4° C for a refrigerator, for example.

NEWS BRIEFS

Ammonia in groundwater: the distillation situation

Everybody knows that groundwater samples must be filtered prior to laboratory testing. But did you know that because this step is so effective at removing the interferants that plague the ammonia analysis, filtered groundwater samples do not have to be distilled before they are analyzed? It's true! Because groundwater samples are always filtered prior to analysis, the distillation step is not necessary. This exemption does *not* apply to wastewater, sludges, etc. Questions? Contact Greg Pils at (608) 267-9564, e-mail: *pilsg@dnr.state.wi.us.*

Reactivity, sulfide and cyanide

Laboratories that are certified to do reactivity testing under category 7 (one of the federal hazardous waste characteristics) should also be certified for sulfide and cyanide in category 6. This is similar to the case for TCLP, where laboratories must be certified for both the extraction and the determinative procedures in the appropriate categories. Please note that the releasable cyanide and sulfide test methods (SW-846 7.1.2 and 7.1.3) specify the determinative methods that must be used. If the tests for releasable cyanide and sulfide appear on your certificate without the tests sulfide and cyanide in category 6, please contact Jeff Ripp at (608) 267-0579.

Substances of Concern at Low Levels

It must be spring. The gobbles of strutting tom turkeys echo through the early morning woods, walleye and steelhead are about to begin their annual spawning runs, a new Major League Baseball season is underway, and the Wisconsin Laboratory Certification Program is publishing its annual list of compounds of concern at low levels. This list is published as a reminder that laboratories are required to report all data for these substances down to their limit of detection. All results greater than the LOD, yet less than the LOQ, must be reported and appropriately qualified (consult NR 149 for definitions of the LOD and LOQ). Be aware that some programs may require the results for *all* compounds to be reported down to the LOD, *even if they do not appear on the list*. Check with your clients to determine what reporting requirements apply. Labs may decide to report *all* data to the LOD, thereby avoiding confusion and insuring reporting requirements are always met.

INORGANICS

Metals
Antimony
Beryllium
Cadmium
Lead
Thallium
Mercury
Chromium (Hexavalent)

ORGANICS

Acids/Phenols
Pentachlorophenol (PCP)

Benzidines
Benzidine

Haloethers
Bis(chloromethyl)ether

Nitroaromatics
2,4-Dinitrotoluene
2,6-Dinitrotoluene

ORGANICS

Polynuclear Aromatic Hydrocarbons
Benzo(a)pyrene

Phthalates & Adipates
Di(2-ethylhexyl)phthalate

Nonpurgeable Chlorinated Hydrocarbons
Hexachlorobenzene

Dioxins/Furans
Dioxin

PCBs
Polychlorinated biphenyls

Chlorinated Pesticides
DDT and Metabolites
Heptachlor
Heptachlor epoxide
Lindane
Toxaphene

ORGANICS

Carbamate Pesticides
Aldicarb

Nitrogen Pesticides
Alachlor
Dimethoate
Parathion
Trifluralin

Volatiles
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,3-Dichloropropene (cis/trans)
Bromodichloromethane
Bromoform
Bromomethane
Chloroform
Chloromethane
Methyl tert-butyl ether (MTBE)
Methylene Chloride
Vinyl Chloride
Dibromochloropropane (DBCP)
Ethylene dibromide (EDB)

UPDATE: FEDERAL REGULATIONS

Diane Drinkman, Lab Certification Program

Pulp, Paper, and Paperboard "Cluster Rule." The combined air and water "cluster rule" for the pulp and paper industry protects human health and the environment by reducing toxic pollutant releases to the air and water. The technology standard in the rule cuts toxic air pollutants by almost 60% from current levels and virtually eliminates all dioxin discharged from pulp, paper and paperboard mills into rivers and other surface waters. Included in the cluster rule are analytical methods for adsorbable organic halides and chlorinated phenolics in wastewater (Methods 1650 and 1653, 40 CFR, Part 430, Appendix A).

EPA Position Statement on Environment Management Systems and ISO 14001. On March 12, 1998, EPA issued a position statement regarding environmental management systems, including those based on the International Organization for Standardization (ISO) 14001 standard. "EPA supports and will help promote the development and use of EMSs, including those based on the ISO 14001 standard, that help an organization achieve its environmental obligations and broader environmental performance goals." The agency collected comments through early April and will be holding stakeholder meetings in the future.

Performance-Based Measurement System Update. In the October 6, 1997, Federal Register, EPA announced plans to implement a performance-based measurement system (PBMS) for environmental monitoring in all of its media programs to the extent feasible. Where PBMS is implemented, the regulated community would be able to select any appropriate analytical test method for use in complying with EPA's regulations. It is EPA's intent that implementation of PBMS have the overall effect of improving data quality and encouraging advancement of analytical technologies. The agency anticipates proposing amendments to its regulations to incorporate PBMS into its regulatory programs.

Hexane Extractable Materials (Method 1664). In January 1996, EPA proposed Method 1664, Hexane Extractable Material, to reduce the demand for chlorofluorocarbons by laboratories. Additional analytical data collected by the agency was made available for public review and comment in October 1996. The latest revision of the method is anticipated to be finalized in 1998.

Drinking Water Contaminant Candidate List Updated. Reauthorization of the Safe Drinking Water Act requires EPA to publish a list of contaminants that are known or suspected to occur in public water systems, and which may require regulation under SDWA. The unregulated contaminants considered for the list include, but are not limited to substances referred to the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA). The final contaminant candidate list is divided among three categories: (1) priorities for drinking water research, (2) additional data collection and (3) consideration for regulation by August 2001. See the March 2, 1998, Federal Register contaminant candidate list.

National certification moves forward in Wisconsin

Alfredo Sotomayor, Lab Certification Program

The third interim meeting of the National Environmental Laboratory Accreditation Conference (NELAC) took place on January 13 -16, 1998 in Washington, D.C. At the interim meeting NELAC committees discussed possible changes to the standards. These changes will be presented at the annual meeting for a vote by the full conference. The Board of Directors was excited to report that at the time of the meeting 18 states had applied for national recognition. As of this writing, 20 state programs have applied for recognition.

A summary of a survey conducted of all of the states was also presented. This survey asked the states to estimate their potential timeline for becoming NELAC accrediting authorities. Wisconsin's position was misrepresented. The information presented at the interim meeting places Wisconsin among the states that will apply for recognition before October 1998. This is not true. In our response, we indicated that **if** Wisconsin does apply for recognition, it would be **after** October 1998.

Closer to home, DNR has authorized a technical advisory committee (TAC) to advise the Department on how to proceed with NELAC adoption. The goal of the TAC is to provide recommendations on whether the Wisconsin DNR should become an accrediting authority recognized by the National Environmental Laboratory Accreditation Program (NELAP) and whether all, some, or none, of the laboratories currently certified or registered in Wisconsin will be included under the NELAC umbrella.

The advisory committee is expected to present recommendations on adoption by July of 1998. The TAC has met twice already and has established a general workplan. Meetings are in Madison and are being facilitated by the DNR. Three subcommittees have been formed to explore adoption questions: one to deal with fiscal implications, another to evaluate NELAC's Quality Systems standard, and a final one to consider the remaining NELAC standards.

Although the final decision on this matter rests with the Department, we hope that by seeking input from all possible affected parties we will make the most equitable and beneficial decision for constituents and for ourselves, the ultimate recipients of regulatory data.

All NELAC advisory committee meetings are open to the public. Our web site will have a page devoted to the NELAC TAC. Please consult our site for a list of the TAC members and their constituency, meeting agenda, and committee minutes.

http://www.dnr.state.wi.us/org/es/science/lc/tac

Here are some of the other highlights from the NELAC interim meeting:

- The teams that will be reviewing prospective accrediting authorities will consist of the NELAP Director, an EPA Region representative, and a state representative. EPA Region II will devise a checklist for auditing accrediting authorities.
- The first laboratories are scheduled to be audited in 1999.
- The training courses for laboratory assessors will be developed by the private sector. A syllabus for course providers is being developed.

(Please see NELAC on page 18)

NELAC, from page 17

- There continues to be increased commitment from EPA in favor of NELAP. The NELAC Board of Directors met with two EPA Assistant Administrators.
- The assessor's manual was not finalized before the interim meeting but will be available in final form for the next annual meeting on June 30. It is likely that the manual will be proposed as an appendix to the On-Site Assessment Standard.
- New language on the "responsible party of record," now called the "technical director, however named" will be up for vote in June.
- A "Sampling and Field Measurements" Standing Committee will be proposed for sanctioning by the conference in June. ELAB and others were interested in this proposal.
- The definition of mobile laboratories was clarified. Up for a vote in June is the consideration that mobile laboratories spending more than three months on the same site will be subject to separate on-site assessments.
- The two-year extension given to accrediting authorities to pass enabling legislation when they otherwise comply fully with the NELAC standards is scheduled to sunset by July 1, 2000.
- The Performance-Based Measurement Systems (PBMS) provisions of the Quality Systems Standard will be proposed for ratification in June.

For more information about NELAC, contact Alfredo Sotomayor at (608) 266-9257 or any member of the NELAC advisory committee.

NR solid waste recycling demonstration grant program

Every year the Department of Natural Resources has $1 million available in matching grant money to fund waste reduction, reuse, and recycling projects. Grantees, including Wisconsin counties, municipalities, public entities, businesses, non-profit organizations and schools, can receive $150,000 or 50% of total eligible costs (whichever is less) for innovative demonstration scale recycling or waste reduction projects.

New this year is a higher funding rate for community-wide projects that focus on decreasing waste at the source. Rather than emphasize the recycling of materials, these projects must target waste reduction and reuse of solid waste generated at area industries, institutions and/or residences. To encourage these activities, selected community-wide waste reduction projects may now receive 75% of total eligible costs.

A recently funded community-wide waste reduction project in Waukesha County involves over 33 organizations, including businesses, non-profit agencies, and local governments. These groups formed a coalition to conduct a regional waste reduction campaign in the Waukesha County area. Participating organizations will conduct workshops, promotional campaigns, and recognition programs to effectively teach the public about waste reduction in all aspects of their lives.

If you have an idea for a **community-wide waste reduction project** or an innovative demonstration scale recycling project, contact Sheila Henneger of the Wisconsin Department of Natural Resources at (608) 266-9426. **Applications for the next grant cycle must be postmarked by August 3, 1998.**

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